IS 11374 (1985): Methods for determination of monocrotophos residues in foods [FAD 1: Pesticides and Pesticides Residue Analysis]

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IS : 11374 - 1985
(Reaffirmed 2000)

Indian Standard

METHOD FOR DETERMINATION OF MONOCROTOPHOS RESIDUES IN FOODS

UDC 664 : 543 : 632·95·028 MONOCROTOPHOS

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 1986

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Indian Standard

METHOD FOR DETERMINATION OF MONOCROTOPHOS RESIDUES IN FOODS

Pesticides Residue Analysis Sectional Committee, AFDC 56

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METHOD FOR DETERMINATION OF MONOCROTOPHOS RESIDUES IN FOODS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 24 June 1985, after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 Monocrotophos formulations are used in agriculture for the control of various insects pests. Its frequent and increased use often results in harmful effects due to toxic nature of residues. Careful assessment of residues is, therefore, an important step in safeguarding human health and in the establishment of regulatory policy.

0.3 In the preparation of this standard, due consideration has been given to the limits of monocrotophos residues which are likely to be laid down under the provisions of *Prevention of Food Adulteration Act*, 1954 and rules framed thereunder, and the specified test methods are sensitive to the levels indicated in **1.1.1**.

0.4 This standard will enable the health authorities and other to follow uniform test procedure for the estimation of monocrotophos residues in various foods.

0.5 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes gas chromatographic method (GLC) for the determination of monocrotophos (3-hydroxy-*N*-methylcoroton amide dimethyl phosphate) residues in foods.

1.1.1 The minimum limit of determination of monocrotophos residue is 0·02 µg/g (0·02 ppm).

*Rules for rounding off numerical values (*revised*).

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

3. SAMPLING

3.1 The representative samples of the material for determining monocrotophos residues shall be drawn in accordance with IS : 11380 (Part 1)-1985†.

4. EXTRACTION

4.1 Apparatus

4.1.1 *Waring Blender*

4.1.2 *Kuderna Danish Concentrator*

4.1.3 *Chromatographic Column* — 25 cm long and 2 cm I.D.

4.2 Reagents

4.2.1 *Acetone* — Glass redistilled.

4.2.2 *Chloroform* — Glass redistilled.

4.2.3 *Adsorbent Mixture* — Charcoal, celite, magnesium oxide (2 : 2 : 1).

4.2.4 *Sodium Sulphate* — Anhydrous.

4.3 Fruits and Vegetables

4.3.1 Weigh 50 g of chopped fruits or vegetables into a waring blender jar and blend the material for 2 minutes with acetone maintaining at least ratio (2 : 1) of solvent to the material. Filter the pulp under suction on buchner funnel containing Whatman No. 1 filter paper covered with a thin coat of hyflo super-cel attached to a 500-ml flask. Apply vacuum, cautiously, until all the solvent has filtered into the flask. Re-extract the pulp with 100 ml portion of acetone and filter the extracts. Wash the pulp thrice with acetone.

*Specification for water for general laboratory use (*second revision*).

†Methods of sampling for the determination of pesticide residues: Part 1 Agricultural and food commodities.

4.3.2 Transfer the extract to Kuderna/Danish concentrator and distil off the solvent as far as possible by adding a drop of propylene glycol. Transfer the material into the separating funnel, quantitatively using acetone. Add 50 ml of saturated sodium chloride solution and extract with chloroform thrice using 50 ml portions each time. Concentrate the chloroform layer to 10 ml in Kuderna/Danish concentrator after drying through anhydrous sodium sulphate.

4.4 Oilseeds, Nuts, Grains — Follow the procedure as described in **4.3.1** and **4.3.2**. Evaporate the chloroform extract to dryness. Dissolve the residue in 50 ml hexane, add 50 ml of acetonitrile in the flask and transfer the mixture to a 500-ml separating funnel. Shake the funnel for 1 minute. Allow the phases to separate and draw off the acetonitrile (lower layer) into a 500-ml round bottomed flask. Extract the hexane layer with an additional 50 ml acetonitrile. Combine the acetonitrile phases and concentrate to dryness. Dissolve the residue in chloroform and make up the volume to 10 ml.

5. CLEAN-UP

5.1 A suitable aliquot of the final extract from **4.3.2** or **4.4.1** is adsorbed on the chromatographic column containing 5 cm layer of anhydrous sodium sulphate and 10 g of adsorbent mixture overlaid on a cotton plug. The column is eluted with 100 ml of chloroform. The chloroform solution is concentrated to 50 ml in a Kuderna/Danish concentrator after adding a drop of propylene glycol.

5.2 Apparatus

5.2.1 *Gas Chromatograph* — A gas chromatograph equipped with a phosphorus sensitive thermoionic emission detector is operated under the following suggested parameters. These parameters may be varied as per available facilities provided standardization is done.

Column	Glass, 1·5 metre long and 35 mm ID packed with 5 percent OV-101 on 80 to 100 mesh gas chrom Q.
Column oven temperature	210°C
Injection port temperature	220°C
Detector temperature	220°C
Gas flow rates	Nitrogen 20 to 30 ml/min Hydrogen 30 to 40 ml/min Air 300 to 450 ml/min
Recorder chart speed	1 cm/min
Retention time	2·8 min approx.

5.2.2 *Microlitre Syringe* — 2 µl and 10 µl-capacity.

5.3 Reagents

5.3.1 *Ethyl Acetate* — AR grade, glass redistilled.

5.4 Procedure

5.4.1 Evaporate the concentrated residue and dissolve in 10 ml ethyl acetate and inject 2 to 10 µl of this solution into gas chromatograph. Identify the monocrotophos peak by its retention time and measure the peak area.

5.5 Calculations

$$\text{Monocrotophos residues, } \mu g/g \text{ (ppm)} = \frac{A_1 \times V_2 \times V_3 \times C}{A_2 \times V_1 \times M} \times f$$

where

A_1 = peak height/area of the sample;

V_2 = volume, in µl, of standard monocrotophos injected;

V_3 = total volume, in ml, of sample solution;

C = concentration, in µg/g, of standard monocrotophos solution;

$$f = \text{recovery factor} = \frac{100}{\text{percent mean recovery}}$$

A_2 = peak height/area of the monocrotophos standard;

V_1 = volume, in µl, of sample injected; and

M = mass, in g, of sample taken for analysis.

NOTE — Percent means recovery is determined by taking untreated control sample to which known amount of monocrotophos is added and analysed as described above.